BULLETIN

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INSTITUTE NEWS

Award of Institute Medals

The Institute of Metals (Platinum) Medal for 1960 has been awarded to Professor ROBERT FRANKLIN MEHL (Pittsburgh, U.S.A.) in recognition of his outstanding contributions to the science of metals and metallurgical education.

In several ways, Professor Robert F. Mehl has rendered distinguished service to non-ferrous metallurgy. By valuable researches of his own he has markedly extended metallurgical knowledge on a number of subjects, particularly on the constitution of alloys, the theory of age-hardening, crystal structure, and recovery and recrystallization after cold-work. In addition, he has created and organized a productive school of advanced, academic metallurgical research and teaching during his tenure, since 1932, of high offices at the Carnegie Institute of Technology in Pittsburgh. There he has been Director of the Metal Research Laboratories and Professor of Metallurgy, Head of the Department of Metallurgical Engineering and, later, Dean of Graduate Studies.

Professor Mehl's metallurgical interests have not been confined to non-ferrous metals for he has taken general metallurgy as his province. In connection with iron and steel, he has carried out many researches on such topics as heat-treatment and forging operations, but he has also actively assisted manufacturing concerns with advice in their metallurgical difficulties. He is the author of " Metallurgy of Iron and Steel ". This same breadth of interest has been profitably deployed in Professor Mehl's fruitful labours for the development of metallurgical education in America, a field in which he has exerted an outstanding influence. This has not been restricted to his college teaching, but has with at least equal effect been exercised also in the work of national committees that he has led and guided. It has been reflected too in his close association with the activities of the American Society for Metals and of the American Institute of Mining and Metallurgical Engineers. The latter body published his " History of Physical Metallurgy " in 1948. The spirit of an enthusiastic educationist has also illuminated Professor Mehl's formulating influence in connection with the issue by American publishers of suitable text-books on a variety of metallurgical topics.

Professor Mehl has received honorary degrees from five universities and colleges in the Americas. He has also been the recipient of four of the most highly-prized medals appropriate for award to metallurgists in the United States—the latest one being the Gold Medal of the American Society for Engineering Education—and has on several occasions been selected to give the special foundation lectures of leading American Societies. He has over a long period served on a number of advisory and technical committees set up by the Government of the United States.

The Rosenhain Medal for 1960 has been awarded to Professor NORMAN JAMES PETCH (Newcastle-upon-Tyne) in recognition of his outstanding contributions in the field of physical metallurgy.

Professor Petch's careful studies of the structure of martensite, austenite, and cementite have solved the important problem of where the carbon atoms lie in these phases, and his investigations of the processes of fracture have been distinguished by vigorous and quantitative testing of conclusions deduced from theory, and have thrown much light on the relationships between slip and fracture and the role of surface energy in relation to fracture stress. The analysis of yield stress and fracture stress in terms of grain size which he adopted in this work has been widely used as a tool by other workers and is commonly known under his name as a " Petch analysis ". Although most of his own work in the fields cited has been carried out on iron and steel, his methods and the analysis just referred to are evidently generally applicable and constitute a notable contribution to the general field of physical metallurgy.

The W. H. A. Robertson Medal for 1959 and Premium of Fifty Guineas has been awarded to Dr. MAURICE COOK and Mr. E. SWAINSON for their paper on " Arc Melting of Reactive and Refractory Metals " (*J. Inst. Metals*, 1958–59, **87**, 161).

Lecture on " Solidification of Alloys "

A General Meeting of the Institute was held at 17 Belgrave Square, London, S.W.1, on the evening of Wednesday, 10 February, when Dr. W. C. WINEGARD, Associate Professor of Metallurgy in the University of Toronto, lectured on " Solidification of Alloys ". The Chair was taken by Professor H. O'NEILL, Vice-President of the Institute and Chairman of the Metal Physics Committee, under whose auspices the lecture was given.

The lecture was concerned with the structure of the solid formed when pure metals and alloys solidified under various growth conditions. The structures obtained were found to be a function of the amount of supercooling; supercooling could be obtained either by thermal means, such that the liquid was cooler than the solid, i.e. a negative temperature gradient, or by constitutional changes at the solid/liquid interface, which produced supercooling with a positive temperature gradient. The latter type—constitutional supercooling—was the main factor in controlling the structure of alloy ingots and castings.

It had been shown theoretically that the condition for no constitutional supercooling was that $\frac{G}{R} = -m\frac{C_0}{D}\left(\frac{1-k_0}{k_0}\right)$,

Publication Policy

In October 1958 the attention of members was drawn to a recommendation of the Publication Committee that condensation of papers on the constitution of alloy systems was very desirable in the interests of both improved clarity and economy of space in the Journal. *This announcement has led some members to believe that it is proposed to subject such papers to special treatment. This is not, and was not, the Committee's intention, as is shown by the presentation of the constitutional papers published since that time.*

On the other hand, the reports of referees and rapporteurs confirm that most papers, no matter of what nature, could have been more concisely presented. The Editorial Board and the Publication Committee therefore once more urge all prospective authors, in their own interests, to present their material as succinctly as possible; supplementary information of interest to specialists can be deposited in the Joint Library, where it will be available on request.

where G was the temperature gradient in the liquid ahead of the interface, R the rate of solidification, m the slope of the liquidus line, C_0 the concentration of solute, D the diffusion coefficient of the solute in the liquid, and k_0 the distribution coefficient, defined as the ratio of the solute concentration in the solid to that in the liquid with which it was in equilibrium.

When the growth variables G and R were changed to allow a small amount of supercooling, a cellular structure developed. This structure consisted of parallel elements of rod-like form which ran in the direction of freezing. The rods were hexagonal in cross-section, and a decanted interface showed an array of cells much like a honeycomb. The experimental conditions for the formation of the cellular structure had been measured in at least six binary systems, and in every case there was good agreement between theory and experiment.

If the growth conditions were changed to promote more supercooling, i.e. there was an increase in R and/or a decrease in G, the cellular structure changed to a branched dendritic structure. Experiments on five binary systems indicated that a definite relationship existed between the concentration of solute and the values of the growth variables G and R at which dendrites formed. The experimentally derived relationship was that $\frac{C_0}{k_0} = A\frac{G}{R^{\frac{1}{2}}}$ where A was a constant.

The lecturer said that one more thing of extreme practical significance could occur as the result of constitutional supercooling and that was nucleation ahead of the interface in the supercooled region. This had been suggested several years ago and verified recently in the case of the aluminium–magnesium system. The onset of equi-axed dendritic growth was found to be dependent upon the concentration of solute and the growth variables in much the same way that columnar dendritic growth depended upon the variables. This led to the possibility of controlling the structure of a casting by controlling the amount of nucleation that occurred ahead of the interface. If no nucleation occurred, then a wholly columnar casting would result, but if nucleation occurred throughout the liquid at one time, the casting could not be properly fed. The answer to good casting control was to control the extent of constitutional supercooling and the nucleating agent, such that nucleation occurred just ahead of the main interface.

Election of Members

The following 18 Ordinary Members, 3 Junior Members, and 5 Student Members were elected on 3 February 1960:

As Ordinary Members

BOURNEZ, Lucien Robert, Ing., Directeur Commercial, Société Nouvelle SPIDEM, Paris, France.

COLLARD, John Cedric, B.A., Metallurgist, Northern Aluminium Co., Ltd., Banbury.

DUNLEAVY, Joseph G., B.S., Battelle Memorial Institute, Columbus, O., U.S.A.

GRAY, Alan Ernest, A.R.I.C., Chief Metallurgist, Centurian Wire Die Co., Ltd., Altrincham.

JOSKEY, Ronald Cecil, Metals Development Technician, Metals Development Laboratory, General Electric Co., Ltd., North Wembley.

KAT, Dirk, Managing Director, Röntgen Technische Dienst N.V., Rotterdam, Netherlands.

LAW, Cyril John, Development Technician, Metals Development Laboratory, General Electric Co., Ltd., North Wembley.

LLOYD, Edward Harry, A.C.T. (Birm.), A.I.M., Editor, *Sheet Metal Industries*, London.

MASON, George Leslie, A.Met., Spectrographer, Research and Development Department, United Steel Companies, Ltd., Rotherham.

MEIER, Herbert, Dipl.Ing., Technical Manager, Britannia Iron and Steel Works, Ltd., Bedford.

MOUDRY, Gilbert A., Technical Director, Harvey Aluminium, Torrance, Calif., U.S.A.

MUELLER, Charles Paul, B.S., Universal-Cyclops Steel Corp., Refractories Division, Bridgeville, Pa., U.S.A.

SAKUI, *Professor* Seita, D.Eng., Department of Metallurgy, Tokyo Institute of Technology, Tokyo, Japan.

SANSONETTI, S. J., B.S., Assistant Director of Research, Richmond, Va., U.S.A.

SAXENA, B. K., B.Sc., Lecturer in Metallurgy, Government College of Engineering and Technology, Raipur (M.P.), India.

SIMONSSON, Hans Lennart, Civ.ing., Överdirektor, Statens Provningsanstalt, Stockholm, Sweden.

WHITING, Arthur Noel, A.I.M., General Manager, Steel Processes Division, Davy and United Engineering Co., Ltd., Sheffield.

WILSON, Norman A., B.S., Manager of Research, Morgan Construction Co., Worcester, Mass., U.S.A.

As Junior Members

BALDWIN, Ian Stanley, L.I.M., Junior Metallurgist, British Non-Ferrous Metals Research Association, London.

SQUIRES, Ian Frederick, B.Met., British Welding Research Association, Abington.

STARR, Trevor Frederick, A.I.M., Physical Metallurgist, Standard Telephones and Cables, Ltd., Paignton.

As Student Members

HARRIS, Rodney, A.Met., L.I.M., Undergraduate, Department of Metallurgy, University of Sheffield.

IRANI, Jamshed Jijibhoy, M.Sc., Post-Graduate Student, Department of Metallurgy, University of Sheffield.

MCQUEEN, Hugh, B.Sc., B.Eng., B.S., Graduate Student, Department of Metallurgy, University of Notre Dame, Ind., U.S.A.

PARMEE, Anthony Charles, Laboratory Assistant, British Non-Ferrous Metals Research Association, London.

TWIGG, Peter Lindsay, B.Met., Research Student, Department of Metallurgy, University of Sheffield.

THE APPLICATION OF THIN-FILM TECHNIQUES TO THE ELECTRON-MICROSCOPIC EXAMINATION OF METALS

As already reported in the Bulletin (*November issue, p.* 13), *a successful Symposium, organized by the Metal Physics Committee, was held in London on* 12 *November* 1959. *Summaries of the discussion at the morning and afternoon sessions, prepared at the request of the Committee, are given below.*

Morning Session

Reported by R. E. Smallman

Dr. N. P. Allen occupied the Chair and the following papers were presented:

"An Outline of the Theory of Diffraction Contrast Observed at Dislocations and Other Defects in Thin Crystals Examined by Transmission Electron Microscopy." By M. J. Whelan.

"Observations of Dislocations in Metals by Transmission Electron Microscopy." By P. B. Hirsch.

"The Observation of Anti-Phase Boundaries during the Transition from CuAu I to CuAu II." By D. W. Pashley and A. E. B. Presland.

Dr. B. A. Bilby (*Metallurgy Department, Sheffield University*) asked whether, if the temperature rise in thin films was small and the observed behaviour was due to carbon deposition, Dr. Hirsch would care to suggest a mechanism.

Dr. P. B. Hirsch (*Cavendish Laboratory, Cambridge*) replied that thermal stress was still a contributory factor though it was a fact that dislocations always moved more rapidly when contamination had built up. It was not yet known, however, how this produced a stress.

Dr. Fisher suggested that the carbon was possibly of importance in reducing the oxide film that was known to hold the dislocations.

Dr. K. F. Hale (*National Physical Laboratory, Teddington*) reported that Dr. Gale and he had recently studied the temperature and stress distribution in thin metal foils, and had concluded that the maximum rise in temperature attained by an iron foil 3000 Å thick in the Elmiskop I (100 kV, 20μ amp, 600 μ condenser apparatus, illuminating beam dia. 4·2 μ) was 75° C. Corresponding maximum temperatures for aluminium and gold would be 30 and 20° C, respectively. From these figures the maximum shear stress was calculated to be 1·8 kg/mm^2, i.e. (μ/1500), which was amply large enough to move dislocations in aluminium.

Dr. D. McLean (*National Physical Laboratory, Teddington*) said that the thickness of the iron foils, as found by tilt-boundary measurements or stereophotograph measurements, was 3000 $\pm$ 500 Å. Accepting the surface-tension values of iron, the maximum shear stress was 1 kg/mm^2, which operated at 45° to the plane and tended to push edge dislocations out of the foil. He asked whether it was possible that most of the edge dislocations had escaped by the time the foil was examined.

In the opinion of Dr. T. Ll. Richards (*Birmingham College of Advanced Technology*), if the bulk properties in work-hardened metals were required, the problem of dislocations had to be treated statistically. The maximum local strain during deformation equalled that caused by a movement of the Burgers vector over three planes (since two planes always registered, it was only when three planes were considered that a stacking fault was visible). The momentum for a jump of a was h/a, where h = distance between three (111) planes. Equating this to the energy/unit volume gave the "technical strength of a metal":

Metal	Calc. (tons/in^2)	Obs. (tons/in^2)
Ni	18	20
Ag	8·4	9
Au	7·2	3·9
Al	7·2	3·8
Cu	14	...

According to Mr. Brandon (*Department of Metallurgy, Cambridge University*) the foil thickness could be obtained from the width of extinction contours, dotted contrast, and surface traces; each of these methods suggested that the foils were ~3000 Å thick.

Dr. J. Nutting (*Department of Metallurgy, Cambridge University*) referred to the question as to whether measurements on thin films were representative of the bulk material. In this connection it was possible to carry out: (*a*) static experiments in order to look at the dislocation distribution; attention had to be paid to the preparation methods, but there appeared to be little damage from the electrochemical technique; or (*b*) dynamic experiments, in which greater care had to be exercised. For example, Dr. Pashley had shown phase changes occurring in films. Nevertheless, in studying phase changes it had to be remembered that there were many more vacancies present than in the bulk material and that these might affect the rate.

Dr. D. W. Pashley (*Tube Investments Research Laboratories, Cambridge*) said that the movement of dislocations in gold was certainly related to the carbon film, since it was necessary to wait ~1 min before movement occurred. Dr. T. Noggle, at Oak Ridge National Laboratories, U.S.A., had suggested that the carbon was in an amorphous form initially and that it was subsequently graphitized by the electron beam, thermal stresses due to the volume contraction being set up at the same time. He asked whether the form of the carbon was known.

Mr. Brandon replied that it was known that oriented paraffinic deposits could be obtained.

Dr. McLean said that if surface-tension values applied in Dr. Pashley's foil, very large stresses indeed would be obtained, and therefore any deposit on the surface would be important.

Dr. Hirsch, commenting on the rise in temperature of ~30° C reported by Dr. Hale and thermal stress >1 kg/mm^2,

said there was still a discrepancy which, as Dr. McLean had pointed out, was due to surface tension. As to edge dislocations moving out of the foil, he said that dislocations were usually in the form of loops that were cut through on thinning. Consequently, the resultant dislocation was left running from top to bottom of the foil, and topologically it was impossible to make these run out.

Dr. W. Webb (*Union Carbide Corp., Niagara Falls, U.S.A.*) said that a poor vacuum usually caused the surface of the specimen to oxidize in the electron microscope, and since irradiation affected the rate of oxidation, it was possible that the electron beam affected the rate of oxidation. Moreover, this oxide layer might act as a source of vacancies, thereby helping climb processes.

Dr. D. Hull, in a communication with Mr. I. Mogford (*A.E.R.E., Harwell*), said that the radiation hardening of steel was produced mainly by increasing the " friction stress " in the lattice to the movement of dislocations, rather than by an increase in the source-locking of the dislocations. The " friction stress " could result from clusters of defects and if of sufficient size might be expected to be visible in the electron microscope.

The main feature of an iron specimen (containing ~0·04% carbon) which had been heated at 900° C. for $\frac{1}{2}$ h and cooled at a rate intermediate between air cooling and furnace cooling and then irradiated at ~60° C for 3 days (~5×10^{16} n.cm^{-2} ~1 MeV), was rings ~300 Å in dia. with a density of 4×10^{14} cm^{-3}. These were similar in appearance to dislocation rings observed in quenched metals and changed their contrast as the specimens were rotated in the microscope. Some of the loops had a black spot in the centre and also loops could be seen edge on as short vertical lines, with contrast similar to dislocations. The plane of the foil in this case was (110). More complicated defects were also observed, such as two concentric rings which changed in contrast as the foil was rotated. Micrographs also illustrated the marked denuded zone around low-angle boundaries. When dislocations were present, they were decorated during the irradiation treatment, often in a complicated pattern.

The damage observed was annealed out rapidly when the foils were heated to 180° C on a hot stage fitted in the electron microscope. The loops without spots in the centre collapsed and disappeared and the loops with the central spot shrank, leaving the black spot, which was not annealed out even after heating to 400° C. The decoration on the dislocations also disappeared at 180° C.

The appearance of the loops in foils of various orientations was consistent with their lying on (011) planes; thus on a (001) foil some loops were seen edge on at right angles to each other as well as approximately circular loops, and in a (011) foil only one set of edge-on loops appeared. However, edge-on loops were not seen in (111) foils even though they were to be expected.

A somewhat similar effect to that produced under irradiation at 60° C was obtained when foils were heated without irradiation, for example, at 60° C for 3 days. There was a much lower density of defects, 4×10^{13} cm^{-3}. The appearance of the defects was often very complicated. It was almost certain that the effects produced without irradiation were due to precipitation of carbon, which had a very low solubility in iron at these temperatures.

The exact nature of the loops produced during irradiation was not yet clear. The higher density of loops in the irradiated material strongly suggested either that the irradiation was aiding the nucleation of carbon precipitation, or that the presence of small agglomerates of carbon provided sinks for point defects formed during irradiation. Alternatively, the point defects might increase the mobility of the carbon solution. If the loops in the irradiated materials were monatomic layers of carbon atoms, it was difficult to explain the contrast effects produced, since there was no evidence of a uniform strain around the precipitate, such as might be expected. However, it was possible for the strain to be relieved by the formation of a dislocation ring round the precipitate which would give the observed contrast. Dislocation loops could also be formed if small carbon agglomerates acted as sinks for point defects, giving interstitial or vacancy platelets. There was strong evidence from the mechanical properties of irradiated iron that the loops served as sinks for point defects.

Dr. R. Philips (*Aeon Laboratory, Englefield Green*) said that micrographs obtained during work done at the A.E.I. Research Laboratories, Aldermaston, on bismuth telluride were remarkably similar to pictures of graphite and mica. Bismuth telluride had only one plane and three directions, which made it a simple system for beginners to study contrast effects on.

Dr. A. Berghezan (*European Research Associates, Brussels*) reported some results of work (by A. Fourdeux and himself) carried out by transmission electron microscopy. These included (*a*) the mechanism of plastic deformation, (*b*) stacking faults in a hexagonal metal (zinc), and (*c*) polygonization in high-purity copper.

To study the mechanism of plastic deformation, a microtensile machine, designed to fit inside the electron microscope, had been constructed. When the method had been applied to aluminium, some new phenomena relating to slip, generation of dislocations, and the role of grain and sub-grain boundaries acting as sources ("donors ") and " acceptors " of dislocations, had been discovered. Among the most important observations were those showing the formation of steps at the surface of single-crystal specimens corresponding to the passage of many hundreds of dislocations. Nucleation of slip, either at the surface or in the interior, had also been observed, and cross-slip appeared very early in the deformation process. This cross-slip assisted the release of the high local stress concentrations which appeared at certain points along the grain boundaries. Inside the crystals, away from grain boundaries, the cross-slip appeared at a later stage and seemed to become frequent as soon as a second slip system became operative. Micrographs indicated that grain boundaries were also important and greatly influenced the deformation; for example, they showed how slip in one grain nucleated slip in a neighbouring grain. The boundaries might also act as sources of dislocations, which propagated either across or along the grain-boundary plane. Another interesting feature of the behaviour of the boundaries was their flow, and photographs had shown that a large amount of deformation was possible in the grain-boundary plane.

In experiments on the stacking faults in zinc, qualitative predictions suggested a high stacking-fault energy and it could not be expected that the stacking faults would be observed by transmission electron microscopy. However, observations showed large dislocation rings, surrounding discs of different contrast, lighter or darker, depending on the orientation. They appeared also in the (0001) plane, which was the orientation of the foil after rolling, and when this plane made an angle with the surface, clear evidence of interference fringes appeared inside them. The loops expanded

readily and frequently during the observation, but the sessile nature of the loops suggested that the mechanism of contraction or expansion was climbing. The origin of the loops was not yet clear, but if it were assumed that they formed by the condensation of vacancies, some mechanism had to operate to create an excess of point defects. Such a possible mechanism might be oxidation of the samples, possibly enhanced by the irradiation with the electron beam.

Experiments on high-purity copper showed that polygonization was possible. The partial dislocations therefore seemed to be not so widely separated as they were generally thought to be. Two restrictions, however, might possibly limit this conclusion: (*a*) a different behaviour of dislocations in thin films from that in the bulk material, and (*b*) diffusion of small amounts of oxygen into the sample, which acted as an impurity and hindered the splitting of the dislocations. Further work was in progress to clarify this problem.

Dr. K. F. Hale (*National Physical Laboratory, Teddington*) reported on some aspects of work on iron (containing carbon $<0{\cdot}05\%$) and its alloys. As far as the electron-microscope observations had gone, the precipitation processes occurring at room temperature and at 100° C were similar, though of course the rate was higher at 100° C. Precipitation was of two types: (1) precipitation on dislocations, and (2) precipitation in the matrix without the aid of dislocations. In low-carbon alloys, i.e. carbon $<0{\cdot}05\%$, precipitation on dislocations predominated. It had been possible in many cases to observe the precipitation on dislocations after various heat-treatments when the plane of the foil was parallel to the (100) plane; the main point of interest in these observations was the dendritic precipitation, along $<100>$ directions, on individual dislocations. The appearance of the micrographs could be explained if it was assumed that the precipitate began by saturating the dislocation and then grew in (100) planes in the form of plates. Thus, if the precipitates were viewed in a [100] direction, the plates would be seen edge on and would appear as orthogonal needles. At other orientations of the foil projected views would be seen, which, if the plates were thin enough, would give image-contrast effects similar to that of an edge dislocation, i.e. a loop appearance at the periphery of the plate. Such precipitates could not be dragged along by a moving dislocation and the dislocation could move under a stress only by tearing away from the dendrite. This mechanism therefore offered a very probable explanation of the yield point in iron.

Precipitation without the aid of dislocations had been observed in a limited number of micrographs. The precipitates were plate-like zones which again lay along (100) planes and were probably coherent with the matrix. When the precipitates became larger, the stresses surrounding them were relieved by the generation of dislocations. These generated dislocations were likely to be responsible for over-ageing; they might also become anchored and could then act as additional barriers. This was probably the explanation of the fact that the creep properties of alloys could be improved by warm working but not by cold working.

The precipitate could also generate dislocation loops in the manner described by Jones and Mitchell in their observations on silver chloride crystals containing embedded glass spheres (*Phil. Mag.*, 1958, **3**, 1). Almost without exception the axes of these loops lay along $<111>$ directions.

Dr. M. J. Whelan (*Cavendish Laboratory, Cambridge*) commenting on the remarks of Dr. Pashley on the need for a high-resolution microscope, said he agreed that this was needed for special cases, such as observation of dislocations in platinum phthalocyanine. Referring to Dr. Berghezan's statement on dislocation loops in zinc, he said that Dr. Brown, at the Cavendish Laboratory, had also examined zinc, but could not obtain the stacking-fault plane at an angle to the foil to observe the contrast. With reference to Dr. Pashley's paper, he said that the contrast across an antiphase domain boundary did constitute an important theoretical case.

Dr. Nutting said that Dr. Berghezan's observations of dislocation movement near grain boundaries suggested that the Taylor criterion for deformation in polycrystals was incorrect. Furthermore, the results might indicate that intercrystalline failure in aluminium at low temperatures was due to the movement of dislocations parallel to the boundary.

Dr. K. Thomas (*National Physical Laboratory, Teddington*) said that studies were being made by the thin-film technique of the dislocation arrays in "equivalent" specimens deformed by tension, creep, or alternating tension. Aluminium creep specimens showed regions with long parallel dislocations. Fatigued aluminium exhibited unusual dislocation arrays, particularly dislocation mazes. These mazes were also found in fatigued gold, and with these specimens slip bands were observed. A major problem was to determine the relative importance of dislocation mazes and slip bands in fatigue. A method of preparing thin films from fracture faces was required.

The thin-film surface technique had been used for further study (with Dr. M. W. Roberts, formerly of the National Chemical Laboratory) of the formation of the oxide layer on aluminium after various heat-treatments. Crystalline oxide had been found to form at the aluminium/amorphous oxide interface at temperatures above 400° C.

Afternoon Session

Reported by R. B. Nicholson and J. Nutting

Professor Sir Lawrence Bragg occupied the Chair and the following papers were presented:

"Electron-Microscopic Studies of Precipitation in Aluminium Alloys." By R. B. Nicholson, G. Thomas, and J. Nutting.

"Electron-Microscopic Observations on the Recrystallization of Nickel." By W. Bollmann.

"The Martensite Transformation in Thin Films of Iron Alloys." By W. Pitsch.

"The Growth, Structure, and Mechanical Properties of Evaporated Metal Films." By G. A. Bassett and D. W. Pashley.

Mr. R. N. Wilson (*Royal Aircraft Establishment, Farnborough*) described a device which enabled thin foil specimens to be strained while under examination in the electron microscope. The straining was achieved by the bending of a bimetallic strip when it was heated electrically. As the arms of the bimetallic strip were large compared with the specimen size, the device behaved as a very hard tensile machine, and was therefore particularly useful for studying fracture mechanisms in thin foils. A ciné film was then presented which had been taken from the fluorescent screen of the electron microscope and which showed how a crack propagated through a pure aluminium foil.

M. Genty and Dr. R. Graf (*O.N.E.R.A., Chatillon-sous-Bagneux, France*) gave some details of their work on Al–Zn–Mg

alloys. The two alloys studied were AZ9G1 (9% Zn, 1% Mg) and AZ7G3 (7% Zn, 3% Mg). The thin foils were prepared by ionic bombardment in the apparatus developed by Castaing. In the first alloy the results were similar to those of Nicholson, Thomas, and Nutting, i.e. small plates of the M' phase were observed lying on the {111} planes of the matrix and some needles lying along [110]. At higher ageing temperatures the transition from M' to M ($MgZn_2$) was detected by X-ray diffraction and by electron microscopy.

However, in the alloy AZ7G3, which was nearer in composition to the alloy studied by Nicholson *et al.*, M. Genty and Dr. Graf failed to detect the M' phase by either technique, and they proposed that the M phase was formed directly. In reply, Dr. NUTTING suggested that this disagreement should not be taken too seriously, since only a few results on the Al–Zn–Mg system were given in the paper under discussion. The complete results were contained in a paper to be published in the *Journal* of the Institute of Metals,* and he believed that a comparison of this paper with the results of Genty and Graf would show a large measure of agreement.

Dr. A. SAULNIER (*Cie. Pechiney, Chambéry, France*), in a communication presented by Professor P. LACOMBE, described some work on the Al–Mg, Al–Mg–Si, and Cu–Be systems. Thin foils were prepared by Saulnier's electropolishing method, mentioned in the review paper by Kelly and Nutting.† Saulnier believed that this method should be adopted on a wider scale, since it was quick and used the standard Disa-Electropol equipment and solutions. The Al–Mg foils showed precipitation of the β′ phase followed by β. In the case of the latter precipitate the (110) planes, whose spacing was 9·9 Å, were clearly resolved. The Mg_2Si precipitates in the Al–Mg–Si alloy were initially in the form of rods lying in many directions, some of them apparently non-crystallographic, but at a later stage a true Widmanstätten pattern was

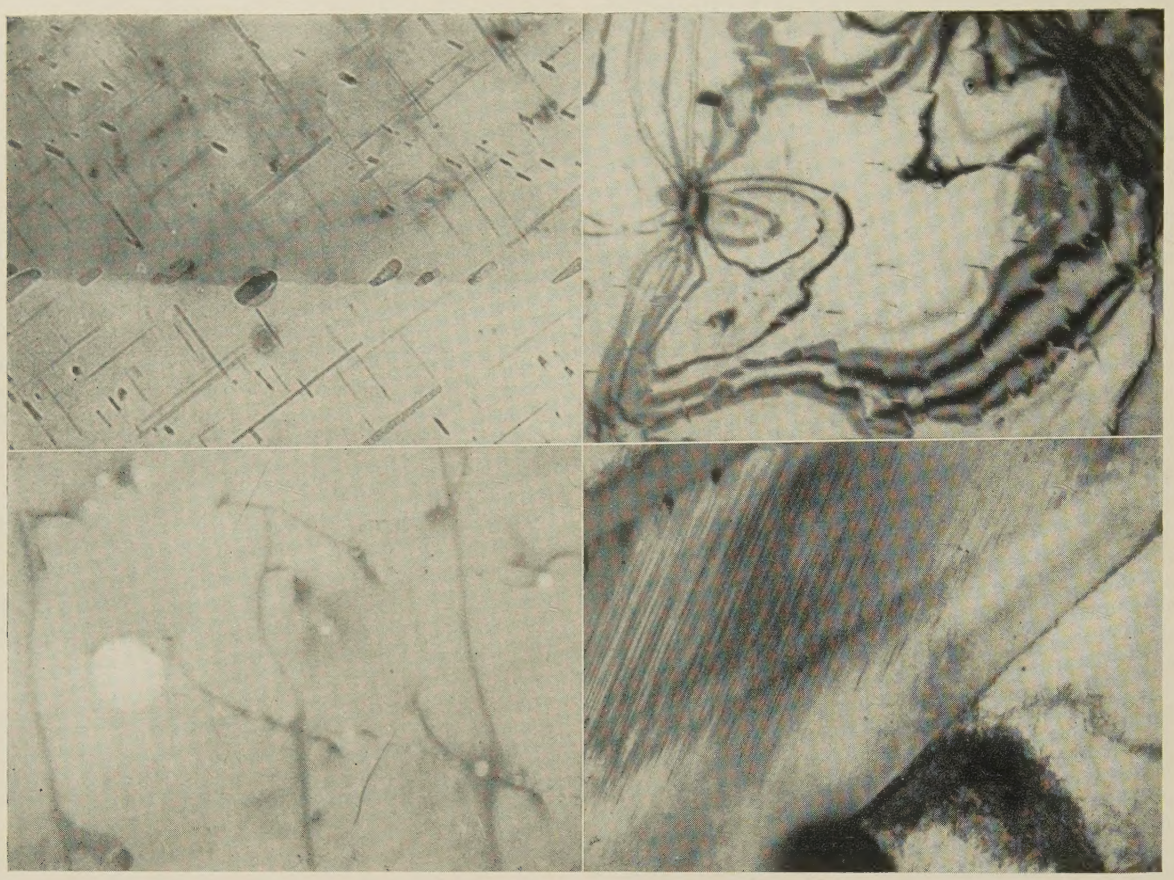

FIG. 1.—Transmission electron micrograph of thin foil from aluminium containing 3·2% Mg_2Si solution-treated and aged for 2 h at 250° C. ×60,000. (*After Saulnier.*)

FIG. 2.—Transmission electron micrograph of thin foil from titanium containing hydrogen. The foil surface is parallel to the basal plane and the hydride precipitates on the {10$\bar{1}$0} planes. ×57,000. (*After Jones.*)

FIG. 3.—Transmission electron micrograph of thin foil of aluminium irradiated with α-particles to produce a concentration of ≃0·02 at.-% helium, and then annealed for 1 h at 550° C. ×60,000. (*After Barnes.*)

FIG. 4.—Transmission electron micrograph of thin foil from 1·0% carbon steel quenched to produce martensite. The narrow bands across the lenticular grain are twins. ×40,000. (*After Kelly.*)

All reduced by one-quarter in reproduction.

* G. Thomas and J. Nutting, *J. Inst. Metals*, 1959–60, **88**, 81.

† P. M. Kelly and J. Nutting, *ibid.*, 1958–59, **87**, 385.

obtained, as shown in Fig. 1. In Cu–Be alloy the precipitate appeared as black irregular shapes.

In reply, Dr. NUTTING suggested that the so-called precipitates in the Cu–Be alloy were etching effects, since any large beryllium-rich precipitate in this alloy would be more likely to appear lighter than the matrix. The possibility of etching was an inherent disadvantage of Saulnier's technique, since some time had to elapse after the disintegration of the specimen before the pieces could be retrieved from the bottom of the container. Dr. Nutting considered that this disadvantage made the method unsuitable for studying some two-phase alloys, although Dr. Saulnier maintained that while the specimens were exposed to the electrolyte very little attack did in fact take place.

Mr. F. O. JONES (*A.E.I. Research Laboratory, Aldermaston*) described the examination of titanium–hydrogen alloys that exhibited very brittle behaviour. He showed that the alloy contained small precipitates of titanium hydride, which occurred in the form of plates on the $\{10\bar{1}0\}$ planes of the matrix. The (100) plane of the hydride was parallel to the plane of the plate. The precipitates gave good diffraction patterns and were made visible by diffraction contrast; a typical micrograph was shown (Fig. 2).

Mr. R. S. BARNES (*A.E.R.E., Harwell*) showed some micrographs of helium precipitation in aluminium irradiated with α-particles. The gas precipitated in the form of bubbles on existing dislocation lines in the matrix, as shown in Fig. 3. In some cases the bubbles were so small that they could be made visible only by de-focusing the microscope, so that the dense diffraction contrast due to the elastic strains near the dislocation did not overwhelm the slight thickness contrast due to the helium bubble. The calculated pressure inside the bubbles was 10^4 atm, but no strain fields were observed near them, and Mr. Barnes suggested that this was due to the fact that the high surface-tension forces acted against this pressure and reduced the elastic strain near the bubbles to a negligible figure.

Mr. P. R. SWANN (*Department of Metallurgy, Cambridge University*) showed micrographs of deformed copper alloys in which regions containing large numbers of stacking faults were either preferentially attacked by the electropolishing solution (e.g. in Cu–Zn alloys) or were more resistant to dissolution than the matrix (e.g. in Cu–Si and Cu–Ge alloys). He suggested that this was due to a Suzuki interaction between the solute atoms and the stacking faults, which resulted in a segregation of the solute to these regions. Mr. Swann considered that such an inhomogeneous distribution of solute would lead to nucleation of surface cracks in corrosive conditions and might explain the high susceptibility of these alloys to failure by stress-corrosion.

In discussing the paper by Bollmann, Dr. J. BAILEY (*Cavendish Laboratory, Cambridge*) reported results which he had obtained on the deformation and subsequent recrystallization of silver. In general, his results were in agreement with those of Bollmann after large amounts of deformation; at smaller deformations, however, he found that the existing grain boundaries were not changed, but that dislocations collected there and recrystallization then started from these regions.

Some interesting new results on recrystallization were presented by M. DIMITROV (*C.N.R.S., Paris*). He had investigated the reheating of 99·97% aluminium which had been deformed 98% and then electrochemically thinned to give foil. He had found that under these conditions the aluminium would not recrystallize even after heating to 600° C; however, sub-boundaries were formed. When the foil thickness was increased to 5 μ, recrystallization occurred at 600° C. When the experiment was repeated with zone-refined aluminium containing 10 ppm of impurity, recrystallization occurred at −80° C in bulk material; with foils <1500 Å thick recrystallization would not take place, however.

In presenting his paper on martensitic transformations, Dr. W. PITSCH (*Max-Planck-Institut für Eisenforschung Düsseldorf*) pointed out how the results he had obtained depended upon the foils being able to relax readily in one dimension, and he therefore predicted that in bulk material the martensitic transformation would occur in a different manner.

Confirmation of this prediction was given in a contribution from Mr. P. M. KELLY (*Department of Metallurgy, Cambridge University*). He had obtained thin foils from steels quenched in the form of plates $\frac{1}{16}$ in. thick and then electrolytically machined. He had found that in high-carbon steels the transformation strains were accommodated by a twinning shear in which twins ranging in thickness from 15Å to $\simeq 500$ Å were found. An electron micrograph was shown (Fig. 4). In low-carbon steels the structure showed needles of martensite rather than plates, whilst in general twinning was absent.

The difference between the orientation relationships of martensite and the parent austenite when transformation occurred near a free surface or within the bulk was also discussed by Dr. H. WILMAN (*Imperial College, London*). In earlier experiments he had examined by reflection electron diffraction the surface of abraded iron, and had found a martensite pattern which he attributed to the formation of austenite during the heating due to abrasion and its subsequent transformation on cooling. Under these conditions transformations were similar to those observed by Pitsch.

The afternoon's proceedings concluded with the showing of three ciné films obtained by photographing the electron-optical images as seen on the fluorescent screen of the microscope. The first two films were presented by Dr. M. J. WHELAN (*Cavendish Laboratory, Cambridge*) and were concerned with the different forms of dislocation movement in stainless steel and aluminium and the annealing out of dislocation loops by the migration of vacancies. The third film, presented by Dr. G. THOMAS (*Department of Metallurgy, Cambridge University*), showed the precipitation of equilibrium $CuAl_2$ from a supersaturated aluminium–4% copper alloy. In order to study this phase transformation and the annealing of loops, a hot stage had been used in the electron microscope, which enabled the specimen to be solution-treated, quenched, and aged *in situ*.

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